SAMPLE COOLING OBSERVING DEVICE

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- European:

Application number: JP19950319951 19951208

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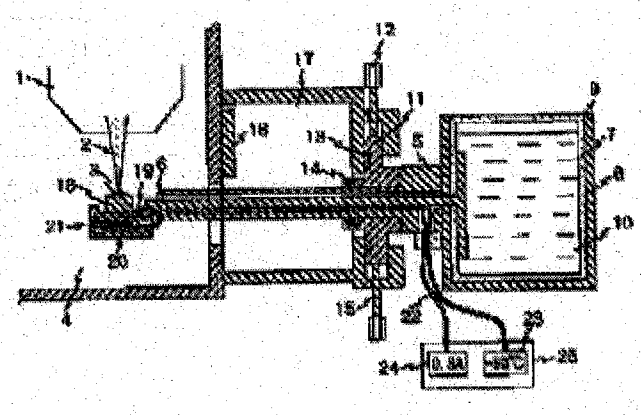
Abstract of **JP9161707**

PROBLEM TO BE SOLVED: To optionally adjust a temperature for many hours up to reaching a sample chamber pressure condition at sample observing time from an atmospheric pressure condition by easily setting and adjusting a low temperature not higher than (-) 20 deg.C. SOLUTION: In a scanning electron microscope having a means to observe a sample 3 by putting it in a cooling condition and a sample cooling observing device of its similar device, a heating means 20 to adjust a sample temperature is arranged in the vicinity of a member on which the sample 3 is place, and the heating means 20 is composed of a member 21 which can optionally heat it up to reaching a sample chamber pressure condition at sample observing time from an atmospheric pressure condition. The heating member 21 is composed of a heating element composed of a platinum wire and an insulating material and a case to shield the heating element, or is composed of a heating element composed of a nichrome wire and an insulating material and a case to shield the heating element.



Family list
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Back to JP916

1 **SAMPLE COOLING OBSERVING DEVICE**

Inventor: OKURA AKIMITSU; SATO MITSUNORI

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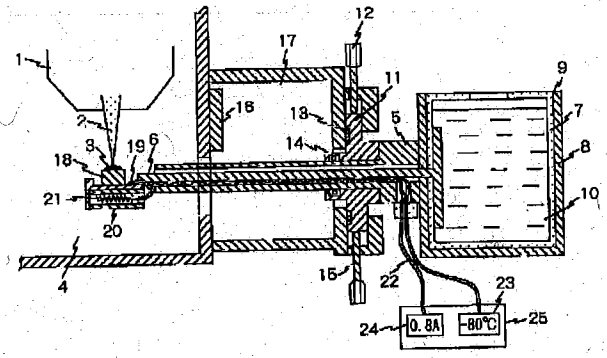
(54)【発明の名称】　試料冷却観察装置

(57)【要約】
【課題】－２０℃以下の低い温度設定や調整が容易で、大気圧状態から試料観察時の試料室圧力状態に至るまで、任意に長時間温度調整が可能な試料冷却観察装置を提供する。
【解決手段】試料３を冷却状態にして観察する手段を有する走査形電子顕微鏡及びその類似装置の試料冷却観察装置において、試料３を載せる部材の近くに、試料温度を調節するための加熱手段２０を設け、加熱手段２０は、大気圧状態から試料観察時の試料室圧力状態に至るまで、任意に加熱できる部材２１で構成する。加熱部材２１は、白金線と絶縁物からなる発熱体と、発熱体を遮蔽するケースとで構成するか、または、ニクロム線と絶縁物からなる発熱体と、発熱体を遮蔽するケースとで構成する。

図　1



【特許請求の範囲】
【請求項１】試料を冷却状態にして観察する手段を有する走査形電子顕微鏡及びその類似装置の試料冷却観察装置において、前記試料を載せる部材の近くに、前記試料の温度を調節するための加熱手段を設け、前記加熱手段は、大気圧状態から前記試料の観察時の試料室圧力状態に至るまで、任意に加熱できる部材で構成したことを特徴とする試料冷却観察装置。
【請求項２】前記加熱部材は、白金線と絶縁物からなる発熱体と、前記発熱体を遮蔽するケースとで構成した請求項１に記載の試料冷却観察装置。
【請求項３】前記加熱部材は、ニクロム線と絶縁物からなる発熱体と、前記発熱体を遮蔽するケースとで構成した請求項１に記載の試料冷却観察装置。
【請求項４】前記加熱部材は、セラミックヒータで構成した請求項１に記載の試料冷却観察装置。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は、走査形電子顕微鏡及びその類似装置に係り、特に、走査形電子顕微鏡に使用するに好適な試料冷却観察装置に関する。
【０００２】
【従来の技術】試料に収束電子線を照射し、走査することにより、試料から得られる信号をもとに走査像を得る装置、いわゆる走査形電子顕微鏡（通称ＳＥＭ）では、試料をできるだけ生の状態で観察したいという要求が最近ますます多くなってきている。そのための方法の一つとして、試料を冷却した状態で観察する、いわゆる試料冷却観察装置が使用されている。また、試料室を１～２７０Ｐａ程度の低真空度にして試料からの水分の蒸発をできるだけ少なくして観察する方法も用いられている。さらに、この両者を組み合わせる方法も用いられている。しかるに、高分子材料や低融点物質など、試料によっては、－２０℃以下の低い温度に素速く冷却し、一定温度に調節しながら観察する場合があり、できるだけ応答性の良い冷却装置が望ましい。また、試料室を１～２７０Ｐａ程度の低真空度にしても、さらには大気圧状態にしても容易に温度調整ができるようになっていることが望ましい。従来技術では、この点での考慮が払われておらず、－２０℃以下の低い温度設定が困難であったり、試料室を１～２７０Ｐａ程度の低真空度にすると、温度調整を行うための加熱部材が焼損により寿命が著しく短くなるなどの難点があった。
【０００３】
【発明が解決しようとする課題】本発明は従来技術の難点をなくし、－２０℃以下の低い温度設定や調整が容易で、かつ大気圧状態でも長時間温度調整が可能な試料冷却観察装置を提供することにある。
【０００４】
【課題を解決するための手段】上記の目的を達成するために、本発明では、試料を冷却状態にして観察する手段を有する走査形電子顕微鏡及びその類似装置の試料冷却観察装置において、前記試料を載せる部材の近くに、試料温度を調節するための加熱手段を設け、前記加熱手段は、大気圧状態から試料観察時の試料室圧力状態に至るまで、任意に加熱できる部材で構成する。前記加熱部材は、白金線と絶縁物からなる発熱体と、前記発熱体を遮蔽するケースとで構成するか、または、ニクロム線と絶縁物からなる発熱体と、前記発熱体を遮蔽するケースとで構成する。あるいは、前記加熱部材は、セラミックヒータで構成した。
【０００５】この構成によれば、－２０℃以下の低い温度設定や調整が容易で、かつ大気圧状態でも長時間温度調整が可能な試料冷却観察装置を提供することが可能となる。
【０００６】
【発明の実施の形態】以下で本発明の具体的実施例を図面を用いて詳述する。
【０００７】図１は本発明の一実施例である。
【０００８】同図において、対物レンズ１によって細く絞られた電子ビーム２は、試料３上で走査され、この試料表面より発生した２次電子や反射電子(いずれも図示せず)が、いわゆるＳＥＭ像としての電子信号となる。試料３は、試料室４に取付けられた試料冷却装置５によって冷却される。すなわち、試料３が載置されている冷却体６の他端は、断熱材７，カバー８などからなる冷却容器９とつながっており、例えば、液体窒素のような冷却剤１０で冷却される。試料の微動は、ステージボデー１１とつまみ１２，真空Ｏリング１３，１４などからなる試料微動装置１５によりＸ，Ｙ，Ｚ方向に移動できるようになっている。また、試料室には、エアロック手段１６を介して試料処理室１７が設けられ、試料の交換や切断などが行えるようになっている。試料の温度制御は、試料台１８の近くに取付けられた熱電対１９と、加熱手段２０によって行う。加熱手段２０は、試料台１８を載せる冷却体６に密着して取付けられた加熱部材２１と、電流導入線２２などで構成されている。温度制御は、試料室の外側より、熱電対メータ２３と、ヒータ電流計２４などから成る温度制御装置２５により行われる。加熱部材２１は、図２に示すように、白金線条２６と絶縁体２７，電流導入端子２８，リード線２９及び遮蔽用ケース３０などから構成されている。遮蔽用ケース３０は、加熱時の光もれ防止と、発熱体からの輻射熱が、試料台１８に効率よく伝わるよう、熱の良導体（金属）で構成され、上面は冷却体６に密着し、下面には真空排気用の孔３１を有している。発熱体に用いている白金線条２６は、空気，水分に対して極めて安定であり、大気中で通電しても、長時間安定に加熱状態を維持できる。また、この構造により、放出ガスが少なく、発熱体は熱しやすく、冷めやすいので、温度制御の応答性も速

いという利点がある。したがって、大気圧条件から試料観察条件（通常、２７０Ｐａ～10^{-3}Ｐａ）の広い範囲にわたって、長時間容易に温度制御が可能である。

【０００９】図３は、本発明における他の一実施例であり、ニクロム線３２を、絶縁体３３の中に封じ込み、外部を遮蔽用ケース３４で囲った加熱部材３５の例である。

【００１０】図４は、本発明における加熱部材の他の一実施例であり、セラミックヒータ３６で構成したものである。この実施例では、内部に発熱体（図示せず）が密封されており、加熱部材そのものを薄く、軽量化できる利点がある。

【００１１】なお、図１では走査形電子顕微鏡に適用した場合についての実施例を示したが、本発明の内容はこれに限定されるものではなく、例えば走査像装置を有する透過形電子顕微鏡に適用した場合についても同様の効果を奏しうる。

【００１２】

【発明の効果】本発明によれば、－２０℃以下の低い温度設定や調整が容易で、かつ大気圧状態でも長時間温度調整が可能な試料冷却観察装置を提供することが可能となる。

【図面の簡単な説明】

【図１】本発明の一実施例を示す断面図。

【図２】本発明の加熱部材の一実施例を示す断面図。

【図３】本発明の加熱部材の第二の実施例を示す断面図。

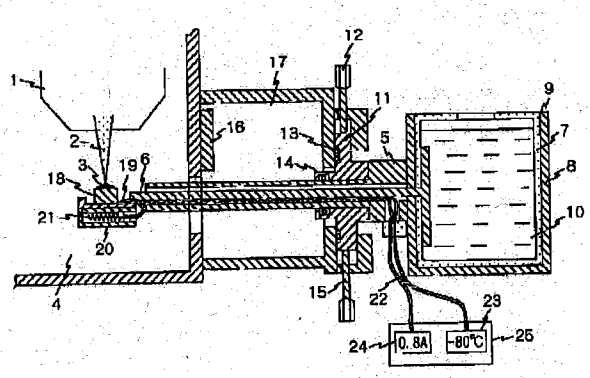
【図４】本発明の加熱部材の第三の実施例を示す断面図。

【符号の説明】

２…電子ビーム、３…試料、６…冷却体、１８…試料台、１９…熱電対、２０…加熱手段、２１…加熱部材。

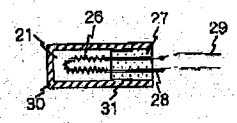
【図１】

図 1



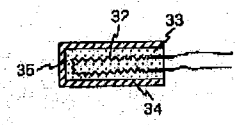
【図２】

図 2



【図３】

図 3



【図４】

図 4

Translation of Patent Publication

Jpn. Pat. Appln. Laid-Open Publication No. 09-161707

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Filing Date: December 8, 1995
Applicants: HITACHI LTD
HITACHI SCIENCE SYSTEMS KK
Inventors: OKURA AKIMITSU
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Priority: Not Claimed
KOKAI Date: June 20, 1997
Request for Examination: Not filed
Int.Cl.: H01J 37/20
B01L 7/00
G01N 1/28
H05B 3/12

[Title of the Invention]

Sample cooling and observing apparatus

[Abstract]

[Object]

To provide a sample cooling and observing apparatus capable of easily setting and controlling a low temperature of -20°C or less and capable of performing prolonged temperature control even under a pressure ranging from atmospheric pressure to sample chamber pressure at the observation time of the sample.

[Means for Achieving the Object]

A sample cooling and observing apparatus for a scanning electron microscope and its similar device, having a means for observing a sample 3 in a cooled state, comprises, near a member on which the sample 3 is placed, heating means 20 for controlling the temperature of the sample 3. The heating means 20 is constituted by a material 21 capable of optionally heating the sample under a pressure ranging from the atmospheric pressure to sample chamber pressure at the observation time of the sample. The heating member 21 comprises a heating element constituted by a platinum wire and an insulator and a case for shielding the heating element or comprises a heating element constituted by a nichrome wire and an insulator and a case for shielding the

heating element.

[What is Claimed is:]

[Claim 1] A sample cooling and observing apparatus for a scanning electron microscope and its similar device, having a means for observing a sample in a cooled state, comprising, near a member on which the sample is placed, heating means for controlling the temperature of the sample, wherein

the heating member is constituted by a material capable of optionally heating the sample under a pressure ranging from the atmospheric pressure to sample chamber pressure at the observation time of the sample.

[Claim 2] The sample cooling and observing apparatus according to claim 1, wherein

the heating member comprises a heating element constituted by a platinum wire and an insulator and a case for shielding the heating element.

[Claim 3] The sample cooling and observing apparatus according to claim 1, wherein

the heating member comprises a heating element constituted by a nichrome wire and an insulator and a case for shielding the heating element.

[Claim 4] The sample cooling and observing apparatus according to claim 1, wherein

the heating member is constituted by a ceramic heater.

[Detailed Description of the Invention]

[0001]

[Technical Field of the Invention]

The present invention relates to a scanning electron microscope and its similar device and, more particularly, to a sample cooling and observing apparatus suitably applied to the scanning electron microscope.

[0002]

[Prior Art]

So-called a scanning electron microscope (SEM), which is a device that irradiates a sample with a focused electron beam to scan the sample so as to obtain a scan image based on a signal obtained from the sample is now increasingly required to allow observation of a sample in a live state as possible. As one method for realizing this, so-called a sample cooling and observing

apparatus for observing a sample in a cooled state is being used. As another method, there is being used a method in which observation is carried out by reducing the pressure in a sample chamber to a low vacuum condition of about 1 to 270 Pa to reduce evaporation from the sample as much as possible. Further, a method combining the above two methods is also being used. Some samples, such as a polymeric material and a low melting point material are required to rapidly be cooled to a low temperature of -20°C or less and be kept at a constant temperature for observation and, thus, it is desirable that the cooling and observing apparatus has a possibly excellent responsiveness. Further, it is desirable that temperature can easily be controlled even if the pressure in a sample chamber is reduced to a low vacuum condition of about 1 to 270 Pa, or even if the pressure of the sample chamber is returned to an atmospheric pressure. The prior arts do not take this issue into particular consideration, and a sample cooling and observing apparatus according to the prior arts has drawbacks that it is difficult to make the setting of a temperature of -20°C or less and that when the pressure in a sample chamber to a low vacuum condition of about 1 to 270 Pa, the life of a heating member for temperature control is significantly reduced due to burnout.

[0003]

[Object(s) of the Invention]

The present invention has been made to eliminate the drawbacks mentioned above and an object of the present invention is to provide a sample cooling and observing apparatus capable of easily setting and controlling a low temperature of -20°C or less and capable of performing prolonged temperature control even under an atmospheric pressure condition.

[0004]

[Means for Achieving the Object(s)]

To achieve the above object, according to an aspect of the present invention, there is provided a sample cooling and observing apparatus for a scanning electron microscope and its similar device, having a means for observing a sample in a cooled state, comprising, near a member on which the sample is placed, heating means for controlling the temperature of the sample, wherein the heating member is constituted by a material capable of optionally heating the sample under a pressure ranging from the atmospheric pressure to sample chamber pressure at the observation time of the sample. The heating

member comprises a heating element constituted by a platinum wire and an insulator and a case for shielding the heating element or comprises heating element constituted by a nichrome wire and an insulator and a case for shielding the heating element, or the heating member is constituted by a ceramic heater.

[0005]

With the above configuration, it is possible to provide a sample cooling and observing apparatus capable of easily setting and controlling a low temperature of -20°C or less and capable of performing prolonged temperature control even under an atmospheric pressure condition.

[0006]

[Embodiment]

A concrete embodiment of the present invention will be described below in detail with reference to the accompanying drawings.

[0007]

FIG. 1 shows an embodiment of the present invention.

[0008]

In FIG. 1, an electron beam 2 focused by an objective lens 1 is scanned on a sample 3, and a secondary electron or reflected electron (which are both not shown), i.e., an electronic signal as so-called a SEM image is generated from the sample surface. The sample 3 is cooled by a sample cooling unit 5 attached to a sample chamber 4. That is, one end of a cooling member 6 on which the sample 3 is placed is connected to a cooling vessel 9 including a heat insulating material 7, a cover 8, and the like and is cooled by a coolant 10 such as liquid nitrogen. The micro movement of the sample 3 can be made in X-, Y-, and Z-directions by a sample micro-movement unit 15 including a stage body 11, a knob 12, a vacuum O-rings 13 and 14, and the like. A sample processing chamber 17 is connected to the sample chamber through an air-lock means 16, whereby replacement or cutoff of the sample can be carried out. The temperature of the sample is controlled by a thermocouple 19 and a hearting means 20 which are attached near a sample stage 18. The heating means 20 is constituted by a heating member 21 firmly attached to the cooling member 6 on which the sample stage 18 is placed, a current lead-in wire 22, and the like. The temperature control is achieved, from outside the sample chamber, by a temperature control unit 25 including a thermocouple meter 23, a heater current

meter 24, and the like. As shown in FIG. 2, the heating member 21 is controlled by a platinum wire 26, an insulator 27, a current lead-in terminal 28, a lead wire 29, a shielding case 30, and the like. The shielding case 30 is formed from a good conductor (metal) of heat so as to prevent light leak at the heating time and achieve effective transfer of radiation heat from a heating element to the sample stage 18. The upper surface of the shielding case 30 is firmly attached to the cooling member 6. A vacuum evacuation hole 31 is formed on the lower surface of the shielding case 30. The platinum wire 26 used as the heating element is extremely stable in the air and moisture and, thus, when being energized in atmosphere, the platinum wire 26 can maintain stable heating properties for a long time. In the above configuration, the amount of emitted gas is suppressed, and the heating element is easily warmed up but just as easily cooled down, so that a high responsiveness for temperature control can be provided. Therefore, it is possible to easily perform prolonged temperature control under a pressure ranging from the atmospheric condition to sample observation condition (generally, 270 Pa to 10^{-3} Pa).

[0009]

FIG. 3 is another embodiment of the present invention, which shows a heating member 35 in which a structure in which a nichrome wire 32 is embedded in an insulator 33 is surrounded by a shielding case 34.

[0010]

FIG. 4 is still another embodiment of the heating member according to the present invention. The heating member of the present embodiment is constituted by a ceramic heater 36. In this embodiment, a heating element (not shown) is hermetically sealed inside the heating member, whereby it is possible to reduce the thickness and weight of the heating member itself.

[0011]

Although FIG. 1 shows the embodiment in which the present invention is applied to a scanning electron microscope, it may be applied to, e.g., a transmission electron microscope with a scanning attachment. Also in this case, the same effect can be obtained.

[0012]

[Advantage of the Invention]

According to the present invention, there can be provided a sample cooling and observing apparatus capable of easily setting and controlling a low

temperature of -20°C or less and capable of performing prolonged temperature control even under an atmospheric pressure condition.

[Brief Description of the Drawings]

[FIG. 1]

A cross-sectional view showing an embodiment of the present invention.

[FIG. 2]

A cross-sectional view showing an embodiment of a heating member according to the present invention.

[FIG. 3]

A cross-sectional view showing a second embodiment of the heating member according to the present invention.

[FIG. 4]

A cross-sectional view showing a third embodiment of the heating member according to the present invention.

[Explanation of Reference Symbols]

2: Electron beam
3: Sample
6: Cooling member
18: Sample stage
19: Thermocouple
20: heating means
21: heating member